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(54) DEPOSITING FILMS OF PLASMA POLYMERIZED MONOMER ON PLASTICS SUBSTRATES

(71) We, THE UNITED STATES OF AMERICA, as represented by The National Aeronautics and Space Administration, N.A.S.A., of Code GP, Washington DC 20546, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to antireflection coatings and methods for their application, and more particularly to anti-reflection coatings for plastics.

Traditionally, glass lenses have been used in cameras, projectors, telescopes, and other optical instruments. Recent developments have shown that lenses can be prepared from thermoplastics by injection molding. The primary advantage of injection molded lenses are low material costs, low weight of finished elements and that only unskilled labor is involved in the manufacturing process since the elements are in a finished state when released from the mold. No milling, grinding, or polishing is required after molding. It is, however, desirable to deposit either a single layer or multilayer antireflection coating on the surface of both glass and plastics lenses to improve the light transmission through the lens. Antireflection coatings reduce the amount of incident light lost upon reflection at the substrate air interface.

Glass lenses are typically coated with a single layer antireflection coating of magnesium fluoride (MgF_2). Magnesium fluoride is deposited on the glass lens by vapor deposition, i.e., by vaporizing magnesium fluoride in a vacuum chamber and then allowing the vapor to contact the heated ($\sim 300^\circ C$) lens. An elevated substrate temperature is required to improve adhesion and durability of the magnesium fluoride coating to the glass surface.

Depositing magnesium fluoride on plastics lenses by vapor deposition is unsatisfactory because thermoplastics generally cannot withstand the high temperatures required for

satisfactory adhesion and durability of magnesium fluoride coating.

Should a "soft" magnesium fluoride coating be deposited on glass then the standard procedure requires baking of the coated element at a temperature between $300^\circ C$. and $500^\circ C$. Plastics suitable for optics are not able to maintain dimensional stability and often oxidize at these temperatures and would be destroyed by the baking process.

The above disadvantages of prior art antireflection coating methods are overcome by the present invention of an improved method for applying an antireflection coating to an optical plastics substrate comprising the step of forming a single layer coating of plasma-polymerized perfluorobutane-2 monomer on the plastics substrate at a temperature less than the softening temperature of the optical plastics substrate.

Prior to coating, the plastics substrates, such as plastics lenses in the preferred embodiment of the invention, are cleaned to remove contaminants and improve adhesion of the antireflection coating. The lenses are next dipped in a first solvent such as Dupont Freon (Registered Trade Mark) solvent TWD 602 to remove water soluble contaminants. The lenses are then rinsed in a second solvent such as Dupont Freon TF to remove the first solvent. The lenses are thereafter vapor degreased in Dupont Freon TF vapor and allowed to dry before insertion into the plasma polymerization reactor. Finally, the lenses are cleaned and activated in the plasma polymerization reactor by an oxygen plasma. Typical oxygen cleaning conditions are 300 seconds in the oxygen plasma at 7×10^{-2} millimeters of mercury, 4 cm³ (STP) per minute flow rate of O₂, and 30 watts of RF power.

It is therefore one object of the present invention to provide a low temperature method for depositing an antireflection coating on an optical plastics substrate;

It is still another object of the invention to provide a method for applying an anti-

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reflection coating simultaneously to both sides of a plastics lens;

It is another object of the invention to provide a low cost method of applying an antireflection coating to a plastics lens;

It is a further object of the invention to provide a method of depositing an anti-reflection coating to a plastics lens irrespective of the lens curvature.

The foregoing and other objectives, features and advantages of the invention will be more readily understood upon consideration of the following detailed description of certain preferred embodiments of the invention, taken in conjunction with the accompanying drawings, which are given by way of example and in which:

Figure 1 is a diagrammatic illustration of a plasma polymerization reactor;

Figure 2 is a graph depicting the rate of deposition of perfluorobutene-2 (PFB-2) on glass coverslips versus power;

Figure 3 is a graph depicting the density of plasma-polymerized PFB-2 coatings deposited at various powers on gold-coated glass coverslips;

Figure 4 is a graph of the transmittance characteristics of a plasma polymerized PFB-2 coated polymethylmethacrylate (PMMA) substrate, for a deposition time of 400 seconds at an RF power of 30 watts with only one side of the substrate being coated;

Figure 5 is a graph of the transmittance characteristics of a PMMA substrate coated with plasma polymerized PFB-2 at 20 and 50 watts of RF power with only one side coated;

Figure 6 is a graph of the transmittance characteristics of a PMMA lens coated with plasma polymerized PFB-2 on both sides for 400 seconds at 30 watts of RF power.

Referring now more particularly to Figure 1, a typical plasma polymerization coating reactor 10 is illustrated as having an enclosed chamber 12, a "hot" electrode 14, a ground electrode 16 spaced from the hot electrode 14, and a substrate holder 18 positioned between the electrodes 14 and 16. The monomer to be deposited on the substrate is fed in through an inlet pipe 20 which exits through a hole in the hot electrode 14. An exit 22 is provided in the chamber 12 and is connected to a trap and vacuum pump 23. The electrode 16 is connected to the circuit ground and to a current probe (not shown). The interior of the chamber 12 is monitored by a pressure gauge 24. The hot electrode 14 is connected to an adjustable RF power supply 26. A substrate 28, for example a plastics lens (which may be composed of polymethylmethacrylate) to be coated, is fixtured midway between the electrodes 14 and 16 which are spaced approximately 2 inches apart. Positioning the substrate between the electrodes, as opposed

to resting it on the lower electrode 16, permits both sides of the substrate 28 to be coated simultaneously. The lens holder 18 is electrically isolated from the lower electrode 16 by legs or other means of support made of an electrically insulating material, such as polytetrafluoroethylene, known under the Registered Trade Mark Teflon. The chamber 12 may be a glass bell jar placed over the electrode assembly. The chamber 12 is evacuated to a background pressure of -10^{-3} mm. of mercury by means of the pump 23.

Monomer vapor, i.e., perfluorobutene-2, is introduced into the bell jar through the monomer inlet pipe 20. The monomer flow rate is kept constant during coating, preferably between 1 and 2 cubic centimeters per minute at STP. Typically, the monomer flow rate is kept constant at approximately 2 cm³/min (STP). Monomer pressure before initiating the plasma is typically 70 microns of Hg, but may for example be between 70 and 150 microns of Hg. A throttling valve 30 is located between the reactor and vacuum pump 23 to minimize monomer consumption during coating by limiting the pumping rate.

High frequency (13.56 MHz) voltage (ranging from 93 to 122 volts peak to peak) is applied across the electrodes by means of the RF supply with impedance matching network 26 to initiate and sustain the plasma. The plasma is very uniform as evidenced by the uniform glow and is confined to an area primarily between the two electrodes 14 and 16. The peak-to-peak current delivered to the plasma during deposition should range from 1.4 to 2.2 amperes at 30 watts.

The deposition is carried out at a substrate temperature which is less than the softening temperature of the substrate, for example not exceeding 40°C. The duration of the deposition is monitored and is used to control film thickness. The plasma is extinguished at the end of a predetermined deposition time by turning off the RF power to the electrodes 14 and 16. Monomer flow is discontinued, the reactor is evacuated to pump out residual monomer, and finally the reactor 10 is back filled with air to break the vacuum. The plastics substrate 28 may then be removed from the reactor 10.

The monomer used in the above described process is perfluorobutene-2 (PFB-2; CF₃—CF=CF—CF₃) yielding a polymer which has a refractive index of 1.39 at a wavelength $\lambda=589.2$ nm. The low refractive index of the plasma polymerized PFB-2 coatings makes them particularly attractive as antireflection coatings for polymethylmethacrylate (PMMA) substrates, which have a refractive index of 1.49 at 589.2 nm.

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The invention is further illustrated by the following non-limiting examples:

EXAMPLE 1

The grounded lower electrode 16 was electrically isolated from the metal support plate 18 to prevent current from flowing through the lower electrode and the support plate. The support plate 18 was in turn isolated from the metal baseplate of the vacuum system, which was further isolated from the ground. All gas flow lines (monomer inlet, vacuum pump, and pressure gauge) were joined with lengths of glass tubing to prevent possible grounding through the instrumentation or the pumping system.

The spacing between the electrodes 14 and 16 was kept at 5.0 cm and the area of each electrode was 182 cm². The samples to be coated were located midway between the electrodes and were supported by an aluminum table with Teflon (Registered Trade Mark) legs. The table rested on the lower electrode 16.

The voltage applied to the upper electrode 14 was measured with a Tektronix (Registered Trade Mark) P6013A voltage probe; the current-to-ground was measured with a Tektronix P6021 current probe. Forward and reflected power were read from the wattmeter incorporated into the 13.56 MHz RF power supply 26.

Reactor pressure was monitored with a thermocouple gauge that was calibrated for PFB-2 by comparison with a McLeod gauge. Gas flow rates were measured with a Hastings linear flow transducer that was calibrated for PFB-2 by comparison with a bubble meter.

Continuous, *in situ* monitoring of temperature was not done during film deposition. However, the temperature of the sample holder was measured immediately after deposition by attaching a thermocouple probe to the sample holder.

The rate of polymer formation under different plasma conditions was determined by weight gain on glass coverslips during a 1200-sec. deposition period. The PFB-2 pressure before turning on the discharge was 9.3 N/m² and the flow rate was 2 cm³ (STP) per minute for all the depositions reported in this application. The density required to calculate the rate in nm/min. from weight gain was obtained from the film physical thickness derived from ellipsometry.

Samples were prepared for the ellipsometer thickness measurements by depositing the PFB-2 on a 4.84 cm² glass coverslip previously coated by vapor deposition on one side with a 10³ nm thick layer of gold. The gold-coated coverslip was weighed on a microbalance before and after polymer film deposition. Film density D was then calculated from the equation D=W/At where

W=film weight, A=film area and t=film physical thickness derived from ellipsometry.

The refractive index of selected plasma polymerized PFB-2 coatings on glass coverslips was calculated from measurements made with a Gaertner Model L119 ellipsometer. The light source was a filtered sodium lamp ($\lambda=589.2$ nm). Samples for the ellipsometer were prepared by depositing thick (>2.5 λ) coatings on glass coverslips. Some refractive index measurements were also made with an Abbe refractometer. The refractive index of plasma polymerized PFB-2 coatings on transparent PMMA substrates was calculated from Brewster's angle which, in turn, was measured with the ellipsometer at the wavelength of a helium-neon laser ($\lambda=623.8$ nm). The more conventional procedure of measuring angles with the ellipsometer to obtain Δ and ψ was not used to measure the coating index because the refractive index of the PMMA substrate was too near the refractive index of the coating. Therefore, it was difficult to obtain a reflection from the coating-substrate interface.

Transmittance spectra in the visible region of plasma polymerized PFB-2 coatings on cast PMMA and uncoated cast PMMA sheet were recorded on a Beckman (Registered Trade Mark) model DK-2A spectrophotometer. Transmittance spectra of PMMA lenses were recorded on a Cary model 14 spectrophotometer. Use of the Cary 14 spectrophotometer to record transmittance spectra of lenses eliminated problems associated with focusing of the incident radiation by the lens on the detector of the spectrophotometer.

Perfluorobutene-2 was supplied by Air Products Co. and was further purified as suggested by the manufacturer to remove nitrogen used in filling the gas cylinder. Thick cast (0.64 cm) PMMA sheet that met MIL-P-5425-C was cut into 2.54 cm squares for use as substrates. Injection-molded PMMA lenses (3.3 cm diameter and ~0.32 cm thick) were supplied by Bell & Howell, Chicago, Illinois 60645.

The PMMA substrates cut from cast PMMA sheet were first cleaned in spirits of gum turpentine to remove the adhesive used to affix a mask to the sheet PMMA, then rinsed in Freon TF (trichlorotrifluoroethane) and finally cleaned in heated Freon TF vapor. Injection-molded PMMA lenses were only cleaned in the Freon TF vapor before coating.

The average rate of deposition (1200-sec. deposition period) of plasma-polymerized PFB-2 on glass coverslips versus power is shown in Figure 2. The density required for calculating the rate in nm/min. from weight gain was obtained from the curve shown in Figure 3. The peak-to-peak voltage applied to the electrodes ranged from 93 to 122 v

- and the peak-to-peak current ranged from 1.4 to 2.2 \AA over the RF power range of 20 to 50 w. The power was restricted to the range from 20 to 50 w (Figure 2) because it was difficult to initiate the plasma below 20 w and the intensity of the glow was nonuniform between the electrodes. Above 50 w the temperature was approaching the softening point of the PMMA substrates. The extrapolated temperature of the sample holder after a 1200-sec. deposition at 50 w was 94°C.
- The decrease in rate of deposition (Figure 2) with increasing power may be ascribed to either temperature or increased rate of polymer degradation with power. Assuming the rate of polymerization is dependent on the number of monomer molecules adsorbed on the glass substrate surface, the temperature is likely to have a significant effect on the rate of polymerization. At low temperature, corresponding to low power, more monomer is adsorbed on the substrate surface than at high temperature. Therefore, the rate of polymerization is higher at low power than at high power. Since the substrate temperature was not controlled in the experiments described here, the heatup time to reach a given temperature is expected to be shorter at higher power. The amount of monomer adsorbed during the heatup time would also be less at higher power and again would lower the rate of polymerization.
- A lower rate of polymerization at higher power may also be due to an increased rate of ablation of the polymer. An increase in bombardment of the polymer by more high-energy electrons and an increase in UV flux from the plasma at higher power may be responsible for an increase in rate of ablation.
- When the coated glass coverslips used for the rate data (Figure 2) were examined for color variations, no interference fringes or other color variations were found across the entire surface of the coverslip. White light from a fluorescent lamp was reflected from the coated surface for this test, and the color of the reflected light was visually examined. This absence of fringes or other color variations is indicative of a coating of uniform optical thickness.
- The decrease in polymer density with increasing power (Figure 3) may be due to fluorine abstraction. It is well known that fluorocarbon polymers prepared by conventional methods have significantly higher densities than most other nonfluorine-containing polymers. By analogy, the loss of fluorine in the plasma polymer is expected to yield a polymer of lower density.
- Deposition rates from Figure 2 were used to calculate the deposition times required to deposit a single-layer antireflection coating on samples of cast PMMA sheet. For minimum reflectance the optical thickness nd of the coating should be one quarter of the average wavelength of the light in which the substrate is to be used. For minimum reflectance at approximately the midpoint (i.e., 550 nm) of the visible spectrum, the optical thickness nd of the coating should equal $\lambda/4$ or 137.5 nm.
- The refractive index n of plasma polymerized PFB-2 coatings deposited at 30 w on PMMA substrates (Samples A and B in Table 1) was found to be 1.37—1.38 at 632.8 nm. Therefore, the physical thickness d required for minimum reflection at 550 nm should be 99.6—100.4 nm. The time required from Figure 2 to deposit at 99.6 nm thick plasma polymerized PFB-2 coating at 30 w was 703 seconds.

EXAMPLE 2

When a single-layer PFB-2 coating was deposited at 30 w on *one side* of a cast PMMA sheet substrate for 703 sec., the transmittance continuously decreased from 700 to 400 nm with no apparent maximum in transmittance (or minimum reflectance). The absence of a maximum in the transmittance curve suggested that the expected optical thickness of the coating was in error. To determine if a change in the optical thickness of the coating would increase the transmittance across the visible portion of the spectrum, coatings were deposited for periods of time shorter than 703 sec. Shorter time periods were chosen because it appeared from the spectrum that maximum transmittance of the 703 sec. coating may be at longer wavelengths, i.e., beyond 700 nm. By depositing coatings for substantially shorter time periods, it was found that significant improvements were made in the transmittance at the shorter wavelengths. Figure 4 shows a transmittance curve for a single-layer coating of PFB-2 deposited for only 400 sec. on a PMMA substrate. It is noteworthy that the transmittance curve of the coated substrate (Figure 4) has the desirable property of being essentially flat over the entire visible range of the spectrum.

The increase in transmittance at shorter wavelengths brought about by decreasing optical thickness of the film suggests that possibly the rate of deposition on PMMA is significantly greater (~1.8 times) than the rate on glass coverslips.

EXAMPLE 3

Figure 5 shows transmittance curves for two PMMA substrates that were coated with plasma polymerized PFB-2 at 20 and 50 w of RF power. The deposition times were 354 sec at 20 w and 520 sec at 50 w of RF power. These deposition times were chosen to yield coatings of equivalent optical thickness, i.e., 56.6 nm physical thickness

based on the rate data in Figure 2. It is evident from Figure 5 that power has only a small effect on transmittance (~0.2%) between 500 and 700 nm. From 400 to 500 nm, the coating deposited at 50 w has a maximum improvement in transmittance of ~0.4% over the 20 w coating. The small improvement in transmittance for the 50 w coating at shorter wavelengths and lower transmittance at longer wavelengths suggests that this coating may be optically thinner than the 20 w coating. The reflected color of the two coatings is also consistent with the 50 w coating being optically thinner.

The reflected color of the 20 w coating was blue, while the reflected color of the 50 w coating was straw yellow. Again, the coatings were visually examined for color variation and none was observed.

The refractive indices of plasma polymerized PFB-2 coatings deposited on PMMA (Samples C and D) at 20 and 50 w were not significantly different (Table I). Also, the similarity in the transmittance spectra (Figure 5) indicates that the refractive indices of the 20 and 50 w coatings are comparable. However, these results do not exclude the possibility that the coatings have an inhomogeneous refractive index.

EXAMPLE 4

Figure 6 is a transmittance spectrum of a plasma polymerized PFB-2 coated lens made of PMMA. The coating was deposited on both sides of the lens simultaneously for 400 sec at 30 w. The transmittance spectrum of an uncoated lens is also shown in Figure 6 for comparison with the coated lens. The significant improvement in transmittance of the PMMA lens resulting from the plasma polymerized PFB-2 coating is clearly evident from Figure 6. The transmittance of the coated lens has increased over the entire visible region of the spectrum and at 550 nm the transmittance improved by 3.4%. Again, there was no evidence of interference fringes or color variation on either side of the lens over the entire surface, indicative of a uniform optical coating. Also, both sides of the lens reflected a straw yellow color when illuminated with white light, which suggests that both sides have coatings of comparable thickness.

A comparison of Figures 5 and 6 shows that the transmittance of the lens near 425 nm does not fall off as rapidly as the transmittance of the coated substrate from cast PMMA sheet. This difference in transmittance may be attributed to the difference in transmittance between the molding pellets from which the lens was prepared and cast PMMA sheet.

Table II gives the transmittance, at

selected wavelengths, of a plasma-polymerized PFB-2 coated PMMA substrate and PMMA substrates coated with other anti-reflection coatings. A magnesium fluoride coating was selected for comparison because it is used extensively for coating glass substrates and has been considered for coating plastic substrates. The multilayer coating (3 layers) was selected because it has been applied successfully to PMMA substrates. Table II shows that the transmittance of the plasma polymerized PFB-2 coated PMMA substrate compares favorably with the other two coatings. Also, at 425 nm, the plasma polymerized PFB-2 coated substrate has a significantly higher transmittance than either the magnesium fluoride or multilayer coated substrates. Extensive environmental testing of the plasma polymerized PFB-2 coated substrates has not yet been completed. However, there has been no evidence of microcracks in the PFB-2 coatings or delamination of the coatings when exposed to ambient air. Magnesium fluoride coatings on PMMA have exhibited microcracks, crazing, and delamination after environmental testing.

While the substrate used in the examples given above was of polymethylmethacrylate, in other embodiments the substrate may be of polystyrene.

Thus we have determined that a suitable antireflection coating may be deposited on a PMMA substrate by plasma polymerization of PFB-2. Although numerous parameters have been given above by way of example, one set of parameters which gives excellent results is as follows:

Substrate: PMMA
 Coating vapor: PFB-2
 PFB-2 vapor pressure in reactor: 70—150 microns of Hg
 Constant flow rate of PFB-2 into reactor: 1 to 2 cc (at STP)/min.
 Duration of plasma generation: 400 seconds
 RF Power: 13.56 MHz, 93—122 volts (peak to peak), 1.4—2.2 amperes (peak to peak), at 30 watts

TABLE I
 Refractive Index at 632.8 nm from Critical Angle Measurements of plasma polymerized PFB-2 Coatings on PMMA Substrates

Sample No.	Refractive Index	Deposition Power, Watts
A	1.381	30
B	1.372	30
C	1.385	20
D	1.373	50

TABLE II
Transmittance at Selected Wavelengths of Uncoated PMMA and PMMA Coated
on One Side With Different Antireflection Coatings

		Wavelength, nm
		425 500 600 700
5	Uncoated PMMA	90.5 91.5 92.5 92.5
	Plasma polymerized PFB-2 coated PMMA	89.5 92.2 93.2 93.0
	Magnesium fluoride coated PMMA	92.8 94.2 94.2 94.2
	Multilayer coating on PMMA	89.0 94.0 94.5 95.0
10	WHAT WE CLAIM IS:—	
	1. A method of depositing a uniform single layer coating on a plastics substrate comprising the step of depositing a film of plasma polymerized monomer onto the surface of the substrate by impinging the substrate with perfluorobutene-2	wherein the coating has an optical thickness of 137.5 nm.
15		5. A method as claimed in claim 2, wherein the substrate is composed of polymethylmethacrylate or polystyrene.
		6. A method as claimed in any one of claims 1 to 5, wherein the substrate temperature does not exceed 40°C.
		7. A method as claimed in claim 2, wherein the plastics substrate is composed of polymethylmethacrylate, the monomer vapor flow rate is between one and two cubic centimeters at STP per minute, the monomer vapor pressure is between 70 and 150 microns of Hg, the RF power is between 93 and 122 peak-to-peak volts, at 13.56 MHz and between 1.4 and 2.2 amperes peak to peak at 30 watts, and the predetermined period of time during which the RF power is applied is 400 seconds.
20	monomer in plasma form, the coating deposition being carried out at a substrate temperature which is less than the softening temperature of the substrate.	8. A method as claimed in claim 2, comprising the steps of positioning the substrate midway between the plasma reactor electrodes, electrically insulating the substrate from at least the lower electrode, and applying the monomer coating during the plasma generation step to both sides of the substrate.
25	2. A method of depositing an anti-reflection coating on a plastics substrate comprising the steps of supporting an optical plastics substrate in a plasma polymerization reactor between the reactor electrodes, evacuating the chamber, introducing perfluorobutene-2	9. A method as claimed in claim 2, comprising the step of first rinsing the substrate in trichlorotrifluoroethane prior to insertion of the substrate into the plasma reactor.
	(CF ₃ —CF=CF—CF ₃)	10. A method as claimed in claim 9, comprising the steps of cleaning and activating the substrate in the reactor before the coating deposition step by introducing oxygen into the plasma reactor and generating an oxygen plasma by applying RF power between the electrodes, the oxygen plasma being so directed as to impinge on the coated substrate surface.
30	monomer vapor into the reactor at a constant flow rate, applying a predetermined RF voltage between the electrodes for a predetermined period of time to generate a plasma stream from the monomer vapor, the plasma stream being so directed as to impinge on at least one surface of the plastics substrate, whereby a coating of plasma polymerized vapor is deposited on the substrate surface, and removing the unreacted monomer vapor and the coated substrate from the chamber at the conclusion of the plasma generation step, the coating deposition being carried out at a substrate temperature which is less than the softening temperature of the substrate.	11. A plastics lens comprising a lens of polymethylmethacrylate and a single layer, antireflection coating on at least one surface of the lens, the coating being plasma polymerized perfluorobutene-2.
35		12. A plastics lens as claimed in claim 11, wherein the antireflection coating is applied to both surfaces of the lens.
40		RONALD VAN BERLYN.
45	3. A method as claimed in claim 2, wherein the step of generating a plasma stream by the application of RF power to the reactor electrodes is carried out for a time sufficient to deposit a coating of plasma polymerized perfluorobutene-2 on the substrate to an optical thickness which is one-quarter of the average wavelength of the light in which the substrate is to be used.	
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55	4. A method as claimed in claim 3,	

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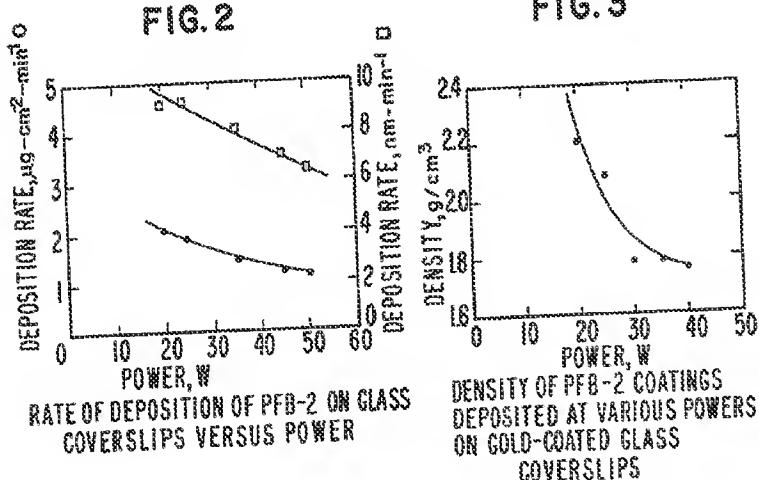
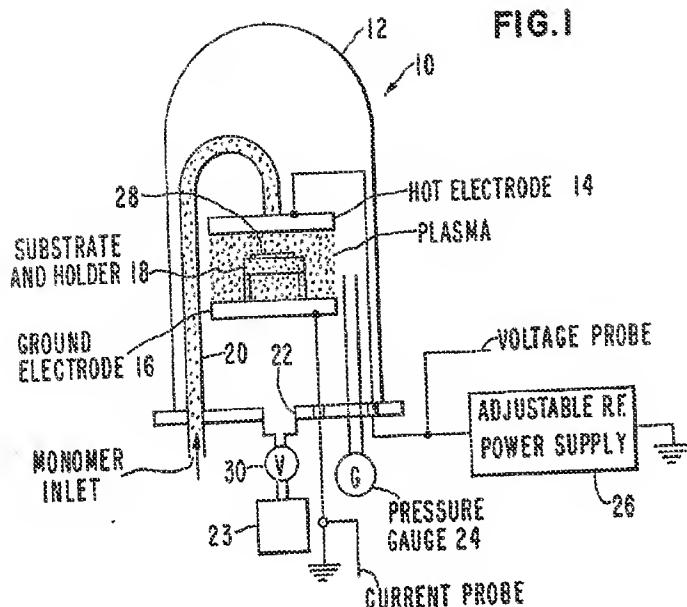
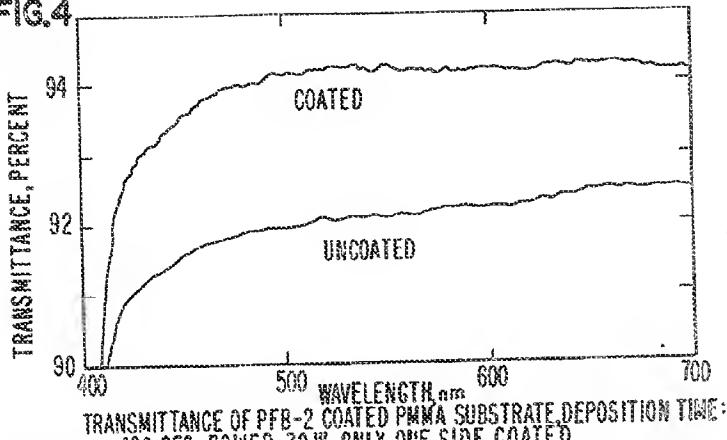
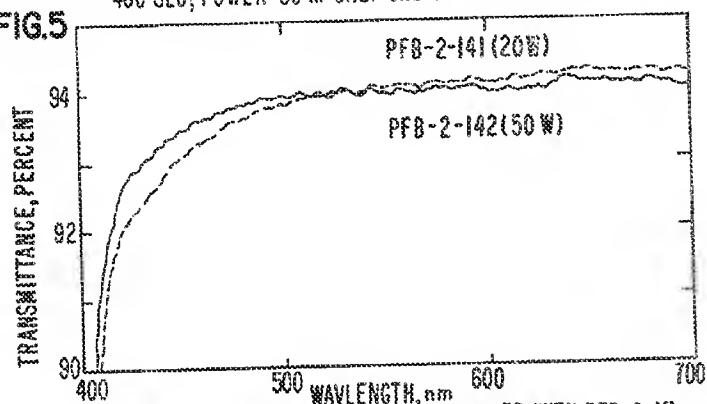


FIG.4



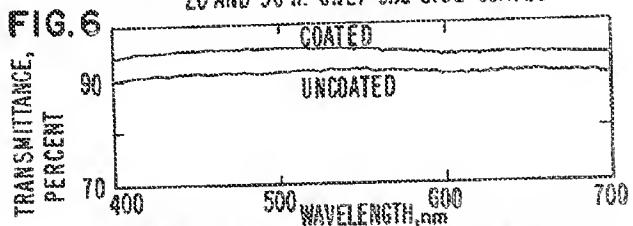
TRANSMITTANCE OF PFB-2 COATED PMMA SUBSTRATE, DEPOSITION TIME:
400 SEC; POWER 30 W. ONLY ONE SIDE COATED

FIG.5



TRANSMITTANCE OF PMMA SUBSTRATE COATED WITH PFB-2 AT
20 AND 50 W. ONLY ONE SIDE COATED

FIG.6



TRANSMITTANCE OF PMMA LENS COATED WITH PFB-2
ON BOTH SIDES FOR 400 SEC AT 30 W